NATIONAL
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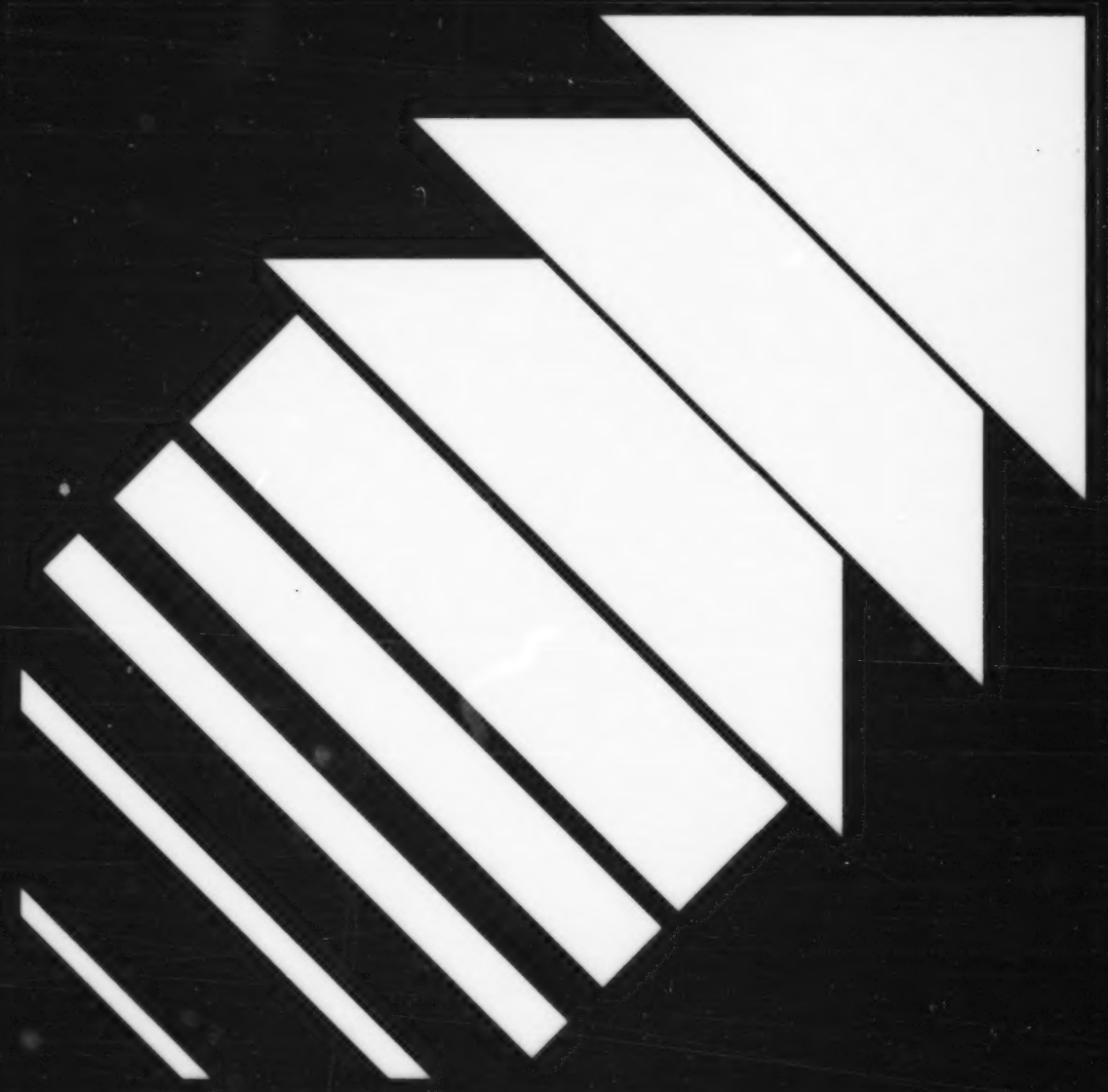
VEGETATIVE RADIONUCLIDE REFERENCE MATERIALS

NUTP-4E



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VEGETATIVE RADIONUCLIDE REFERENCE MATERIALS

J.L. Dalton and W.S. Bowman

National Uranium Tailings Program

NUTP-4E

MICROMEDIA

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Canada Centre for Mineral and Energy Technology

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FOREWORD

In Canada there are currently about 130 million tonnes of mine wastes (tailings) produced from the mining and milling of uranium ore that are on or near the surface. At public hearings and other forums held in connection with proposals for new uranium production facilities, concern has been expressed about the impact that these wastes may have on the environment in the future. Government regulatory agencies believe that there may be long-term environmental and health consequences associated with uranium tailings that are not yet fully understood.

As a result of a year-long study by a group of experts from government, industry and the universities, into the possible long-term problems of uranium tailings management, the Federal Government decided to launch an R&D program to assist in clarifying the nature and extent of problems that may arise in the future. It is anticipated that the results of such a program will be of use to the Atomic Energy Control Board and to provincial regulatory agencies in the establishment of criteria for the decommissioning and abandonment of uranium tailings. To this end, the National Uranium Tailings Program of CANMET has been funded to the extent of $8.5 million for a period of five years.

The main objective of this Program is to provide a scientifically credible information base that will assist government regulatory agencies in making confident, knowledgeable decisions, and in establishing criteria for the long-term protection of the environment and human health. The scientific information base will, among other things, describe the chemical and physical processes that involve tailings constituents and their movement along pathways from the tailings into the biosphere.

The National Uranium Tailings Program's activities, which have been entirely contracted out, have included research into the processes involved in the behaviour and movement of contaminants from tailings, field sampling and measurements, chemical analyses, and predictive modelling. As an important part of the Program output, manuals have been prepared that incorporate the results of this work.

This manual has been prepared for the purpose of making available up-to-date information and data that will be of interest and practical use to those directly concerned with operational aspects of uranium tailings management and regulation.

W.G. Jeffery
Director General
Canada Centre for Mineral and Energy Technology

PREFACE

In late 1983, the Analytical Subcommittee of the Canadian Uranium Producers Metallurgical Committee pointed out to the National Uranium Tailings Program (NUTP) that there was a need for biological analytical reference materials certified for radionuclides of the uranium-decay chain. NUTP agreed to fund the preparation of these materials because they would be useful in the quality assessment of the analysis of materials – not only for NUTP's own projects but also for that of the analytical community at large. Two types of biological materials – vegetative and fish – were collected and suitably prepared. Unfortunately, the concentrations of the radionuclides of interest in the fish-processed fractions were too low to be of practical use. However, the vegetative materials – spruce twigs and needles – were suitable, and this report describes the preparation and ensuing interlaboratory analytical program in the establishment of these two vegetative materials for use as reference materials.

ACKNOWLEDGEMENTS

I would like to thank H.F. Steger, P. Westra, D. McIntosh, C.W. Smith, and R. Sutarno in the preparation and distribution of the materials.

In addition the contribution of the volunteer laboratories involved in the CLV-1 and CLV-2 exchange is gratefully acknowledged. In particular I would like to express a special appreciation to Lloyd Dalton, who acted as scientific authority, and whose efforts ensured the success of this work.

Roy John
Project Manager, Measurements
National Uranium Tailings Program

VEGETATIVE RADIONUCLIDE REFERENCE MATERIALS

J.L. Dalton* and W.S. Bowman**

SYNOPSIS

Two samples, CLV-1 and CLV-2, consisting of spruce twigs and spruce needles respectively, from the Cluff Lake uranium mining area in northern Saskatchewan, were prepared as analytical reference materials. Each material was milled and ground to minus 212 μm, blended, and bottled in 125-g units. The radioactivities of lead-210, polonium-210, radium-226, and thorium-230, and the concentration of uranium were determined in an interlaboratory program involving eight laboratories. Consensus values and associated parameters for the radioactivities were calculated by statistical treatment of the results.

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RADIONUCLÉIDES DE NATURE VÉGÉTALE UTILISÉS COMME MATÉRIAUX DE RÉFÉRENCE

J.L. Dalton* et W.S. Bowman**

RÉSUMÉ

Deux échantillons, le CLV-1 et le CLV-2, consistant dans des brindilles et aiguilles d'épinette, respectivement, et provenant de la région de Cluff Lake, au nord de la Saskatchewan, où des mines d'uranium sont exploitées, ont été préparés comme matériaux de référence analytiques. Chaque échantillon a été broyé à moins 212 µm, mélangé et embouteillé en unités de 125 g. La radioactivité du plomb 210, du polonium 210, du radium 226 et du thorium 230 de même que la concentration de l'uranium ont été déterminées dans le cadre d'un programme interlaboratoire auquel huit laboratoires ont participé. L'analyse statistique des données a été utilisée pour calculer les valeurs reconnues et paramètres associés.

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INTRODUCTION

There is a need for analytical organic reference materials for members of the uranium-decay series in environmental studies on uranium mining and milling. These reference materials will be used as quality-control materials within individual analytical laboratories and in assessing the accuracy and precision of new radiochemical methodology.

The radionuclides of thorium and uranium, radium-226, lead-210, and polonium-210 are of particular interest to the Canadian mineral industry. Whereas some biological reference materials certified for certain radionuclides are available from the National Bureau of Standards (1), there are no "known" biological reference materials certified for the above-mentioned radionuclides. Accordingly, a project was initiated in 1984 under the auspices of the National Uranium Tailings Program to meet this need.

PREPARATION OF REFERENCE MATERIALS

About 200 kg of spruce twigs and needles were collected from the Cluff Lake uranium mining area of northern Saskatchewan. Specifically the material was collected at two sites located adjacent to the current tailings pond and at a third site about 100 m from a worked-out zone of uranium mineralization. The collection was carried out under contract by Dr. C.E. Dunn, biogeochemist, Regina, Saskatchewan, (see Appendix A).

The spruce material was separated into needles and twigs, and then was air-dried, shredded, ball-milled, and blended. The powder-like material was passed through a 212-μm sieve and was bottled in 125-g units which were subsequently sealed in foil pouches to maintain sample integrity. The preparatory work was carried out by the staff of the Canadian Certified Reference Materials Project. The reference materials CLV-1 and CLV-2 represent the spruce twigs and needles fractions, respectively. All of the material was subjected to a batch gamma-ray irradiation to a minimum dose of 25,000 Sv for sterilization purposes.

INTERLABORATORY PROGRAM

Analytical contracts for the analysis of the two reference materials were issued to Chemex Labs Ltd., North Vancouver; to the Saskatchewan Research Council; and to Monenco Analytical Laboratories of Calgary (see Appendix A). The Chemical Laboratory, Mineral Sciences Laboratories, CANMET, and five volunteer laboratories provided additional analytical results. The volunteer laboratories were the Radiochemical Laboratories of the Atomic Energy Control Board, Eldorado Resources, Ottawa, Ontario Hydro, Pickering, the Freshwater Institute, Winnipeg, and the Slowpoke Reactor at the University of Toronto. Participating laboratories are listed alphabetically in Appendix B.

The contract laboratories were provided with one bottle of each material and were instructed to provide four independent results for each of radium-226, lead-210, polonium-210, and thorium-230. The calibration materials used for these determinations were to have a total uncertainty of 5% or less and the counting periods for each determination were to be sufficiently long so that the propagated activity uncertainty would not exceed 5% relative at the one-sigma confidence level.

The volunteer laboratories were asked to conform, if possible, to the instructions given to the contract laboratories. In addition, all laboratories were asked to supply any other analytical results that would be of benefit to the interlaboratory program.

RESULTS OF THE INTERLABORATORY PROGRAM

The interlaboratory program provided analytical data for several elements and radionuclides. However, there were sufficient analytical data to warrant the estimation of consensus values only for uranium, lead-210, polonium-210, radium-226, and thorium-230. The grand mean of all individual determinations, exclusive of statistical outlier sets and/or data points, is defined as the consensus value for the activity of a radionuclide for purposes of this program (2). Consensus values so determined are shown in Table 1. Provisional values, issued for cesium-137 based on three laboratories are shown in Table 2. Table 3 contains various additional analytical data for various elements and radionuclides from only one laboratory and is presented for information purposes only.

The analytical data were inspected visually for obvious outlying results and irregularities. Sets with unusually high variance were subjected to Dixon's test to determine if a single datum point was causing this irregularity (2). Two such cases were found and those data were not used for further computations. A level of significance of $\alpha = 0.05$ was used for

this purpose. Also, three sets of results for radium-226 – the means of which differed by more than twice the overall standard deviation from the mean value initially calculated – were not used in computing the statistical parameters. Table 4 contains the statistical data from the interlaboratory program.

The analytical results obtained from the participating laboratories are presented in Tables 5 to 10. The numerical label of the laboratories in these tables does *not* correspond to their position in the alphabetical list of participating laboratories (Appendix B). Radiochemical methodologies are summarized in Appendix C. The contractors' reports are held on file at the offices of the National Uranium Tailings Program. The calibration materials used by the participating laboratories were recognized solutions and solid powders with total uncertainties of less than 5%.

Many of the laboratories that participated in this program have also participated in other CANMET interlaboratory programs, and a detailed overview of calibration technology in current use may be obtained from the documentation on previous interlaboratory programs (2,3,4).

DISCUSSION

The estimates of precision and accuracy of the consensus values deserve comment. The between-laboratory variance is significant relative to the within-laboratory component, which has been the experience of other interlaboratory programs (2,3,4).

The 95% confidence intervals of lead-210 and radium-226 in CLV-1 are comparable to those obtained for reference material DL-1A in a considerably larger interlaboratory program, even though DL-1A has twice the concentration of CLV-1 (3,4). DL-1A is a powdered, waste-rock material from the Elliot Lake area, completely different from CLV-1. These comparable confidence limits are a measure of the current extent of agreement between participating laboratories at concentrations well above the detection limit and are evidently independent of matrix. The confidence limits for uranium and thorium-230 in CLV-1 are comparable to those of radium-226 and lead-210. However, polonium-210 has a fairly wide confidence interval in both reference materials.

All four laboratories used the same methodology, which has proved reliable in the CANMET interlaboratory program on tailings materials (2), albeit at higher concentrations and for a different matrix.

They all obtained an acceptable within-laboratory standard deviation. The wide confidence interval therefore reflects poor between-laboratory agreement and indicates difficulties in determining polonium-210 at this concentration or matrix, or both.

The concentrations in CLV-2 are lower than in CLV-1 by an order of magnitude or more, and accordingly, the confidence intervals are wider. Even though each datum point submitted by the laboratories was to have a counting uncertainty of 5% or less, agreement between laboratories was not as good as for CLV-1 or as in other interlaboratory programs (2,3,4). These wide confidence limits may serve as a guide in the interpretation of laboratory results in materials of this type at these concentrations.

It should be pointed out that radiochemical equilibrium does not exist in either CLV-1 or CLV-2. This result is to be expected because the uptake of the radionuclides is by an aqueous elemental or radionuclide process, and not by a whole grain or mineral process. Unfortunately, there are analytical ramifications in the lack of radiochemical equilibrium. With respect to CLV-1, polonium-210 is somewhat lower than the concentration of lead-210, but it is expected that these two radionuclides will be in equilibrium by October 1986, which is approximately five half-lives of polonium-210 after the preparation of CLV-1.

In CLV-2, lead-210 and polonium-210 are in equilibrium but radium-226 will not support lead-210. This lack of support between long-lived radionuclides is not expected to be a practical analytical problem because the material will probably be exhausted before these problems become significant. A more serious problem, however, is that of radon emanation from both CLV-1 and CLV-2. Smith found the radon emanation from these materials to be significant: 74% and 30% for CLV-1 and CLV-2, respectively (5).

A practical consequence of the loss of radon is that both lead-210 and polonium-210 concentrations will decrease slightly with time, as shown in Tables 11 and 12, reproduced from the report by Smith (5). Table 12 also shows the expected decline in the cesium-137 activity.

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TABLES

Table 1 – Consensus values for constituents in CLV-1 and CLV-2

Reference material	Constituents (Bq g)				
	Pb-210	Po-210	Ra-226	Th-230	U*
CLV-1	0.66	0.49**	0.70	0.31	86.8
CLV-2	0.074	0.073	0.038	0.033	3.6

* µg g
**As of January 1986, expected to reach the confidence limits of Pb-210 by October 1, 1986, and thereafter remain in radiochemical equilibrium with Pb-210.

Table 2 – Provisional value for cesium-137 in CLV-1 and CLV-2

Reference Material	Cs-137 (Bq g)
CLV-1	0.11
CLV-2	0.034

Three laboratories provided eight determinations for both CLV-1 and CLV-2. The measurements were performed in 1985.

Table 3 – Analytical information on constituents (µg/g unless otherwise indicated)

Constituent	Reference material	
	CLV-1	CLV-2
U-238*	1.06 ± 0.06	0.044 ± 0.002
U-234*	1.06 ± 0.06	0.045 ± 0.002
Th-232*	0.0022 ± 0.0002	0.0006 ± 0.00007
Th	0.53	0.14
K-40*	0.080 ± 0.009	0.12 ± 0.02
Co	≤3.8	≤4.5
Dy	≤0.53	≤1.1
Ba	≤170	≤280
Ti	≤130	≤160
I	≤4.0	≤8.0
Br	≤10	≤20
Mg	≤800	≤1000
Cu	≤40	≤41
Na	280 ± 20	≤70
V	3.6 ± 0.4	≤0.76
Mn	612 ± 7	1940 ± 20
Cl	220 ± 10	440 ± 60
Sr	≤310	≤530
% Ca wt %	0.63 ± 0.03	0.77 ± 0.04
% K wt %	≤0.20	≤0.42
Al**	1430 ± 40	320 ± 20

* Bq g
**Not corrected for any phosphorus contribution in neutron activation analysis determination.

All values in this table are single results.

Table 4 – Consensus values and related statistical parameters, Bq/g

	Radionuclides					
	Consensus value 95% confidence interval (No. sets, No. values, $\bar{C}\bar{V}\%$)					
	Pb-210	Po-210	Ra-226	Th-230	U	U*
CLV-1	0.66	0.49**	0.70	0.31	1.07	87
	0.60-0.72	0.29-0.68	0.67-0.73	0.28-0.33	1.01-1.13	82-91
	(6,23,3.5)	(4,15,6.8)	(7,26,4.2)	(6,23,10)	(8,29,2.4)	(8,29,2.4)
CLV-2	0.074	0.073	0.038	0.033	0.044	3.6
	0.065-0.083	0.052-0.094	0.026-0.050	0.019-0.047	0.038-0.051	3.1-4.2
	(6,23,8.0)	(4,16,7.1)	(7,25,6.9)	(6,22,11)	(8,30,7.5)	(8,30,7.5)

* µg/g

$\bar{C}\bar{V}\%$ = average within-laboratory relative standard deviation.

**At January 1986, expect that Po-210 will grow in and reach the confidence limits of Pb-210 by October 1, 1986, and thereafter be in radiochemical equilibrium with Pb-210.

Table 5 – Laboratory results, means, and standard deviations for lead-210 in CLV-1 and CLV-2 (Bq/g)

Laboratory	Independent results				Mean	S.D.
			Reference Material CLV-1			
LAB- 1	0.75	0.72	0.75	0.72	0.735	0.017
LAB- 2	0.63	0.62	0.63	0.63	0.628	0.005
LAB- 3	0.662	0.729	0.684	0.634	0.677	0.040
LAB- 4 (Beta)	0.66	0.63	0.64	0.61	0.635	0.021
LAB- 4 (Gamma)	0.667	0.765	0.646	0.683	0.690	0.052
LAB- 8	0.575	0.566	0.581		0.574	0.008
			Reference Material CLV-2			
LAB- 1	0.09	0.08	0.08	0.08	0.083	0.005
LAB- 2	0.066	0.065	0.059	0.067	0.064	0.004
LAB- 3	0.092	0.079	0.093	0.062	0.082	0.015
LAB- 4 (Beta)	0.073	0.061	0.063	0.060	0.064	0.006
LAB- 4 (Gamma)	0.0773	0.0825	0.0841	0.0791	0.081	0.003
LAB- 8	0.0754	0.0678	0.0710		0.071	0.004

Table 6 – Laboratory results, means, and standard deviations for polonium-210 in CLV-1 and CLV-2 (Bq/g)

Laboratory	Independent results				Mean	S.D.
			Reference Material CLV-1			
LAB- 1	0.35	0.38	0.36	0.32	0.353	0.025
LAB- 2	0.46	0.45	0.41	0.41	0.433	0.026
LAB- 3	0.664	0.515	0.638	0.582	0.600	0.066
LAB- 8	0.606	0.574	0.581		0.587	0.017
			Reference Material CLV-2			
LAB- 1	0.06	0.06	0.07	0.06	0.063	0.005
LAB- 2	0.065	0.065	0.059	0.066	0.064	0.003
LAB- 3	0.092	0.087	0.099	0.087	0.091	0.006
LAB- 8	0.0764	0.0689	0.0828	0.0682	0.074	0.007

Table 7 – Laboratory results, means, and standard deviations for radium-226 in CLV-1 and CLV-2 (Bq/g)

Laboratory	Independent results				Mean	S.D.
			Reference Material CLV-1			
LAB- 1	0.71	0.70	0.73	0.72	0.715	0.013
LAB- 2	0.70	0.71	0.68	0.65	0.685	0.027
LAB- 3	0.774	0.774	0.673	0.829	0.763	0.065
LAB- 4 (Alpha)	0.690	0.695	0.690	0.682	0.689	0.005
LAB- 4 (Gamma)	0.687	0.689	0.631	0.685	0.673	0.028
LAB- 5 (Alpha)	0.671	0.687	0.742		0.700	0.037
LAB- 5 (Gamma)	0.992	1.05			1.021	0.041
LAB- 8	0.693	0.651	0.721		0.688	0.035
			Reference Material CLV-2			
LAB- 1	0.05	0.06	0.05	0.05	0.053	0.005
LAB- 2 (Alpha)	0.014	0.014	0.013	0.014	0.014	0.001
LAB- 2 ((Gamma)	0.043	0.040			0.042	0.002
LAB- 3	0.043	0.0396	0.0440	0.0436	0.043	0.002
LAB- 4 (Alpha)	0.042	0.032	0.041	0.043	0.040	0.005
LAB- 4 (Gamma)	0.0370	0.0394	0.0373	0.0407	0.039	0.002
LAB- 5 (Alpha)	0.0737	0.0787	0.1017		0.085	0.015
LAB- 5 (Gamma)	0.089	0.095			0.092	0.004
LAB- 8	0.0389	0.0344	0.0400		0.038	0.003

Table 8 – Laboratory results, means, and standard deviations for thorium-230 in CLV-1 and CLV-2 (Bq/g)

Laboratory	Independent results				Mean	S.D.
			Reference Material CLV-1			
LAB- 1	0.32	0.30	0.29	0.33	0.310	0.018
LAB- 2	0.32	0.28	0.30	0.29	0.298	0.017
LAB- 3	0.309	0.349	0.264	0.282	0.301	0.037
LAB- 4	0.30	0.29	0.33	0.46	0.345	0.079
LAB- 6	0.31	0.33	0.33	0.31	0.320	0.012
LAB- 8	0.282	0.228	0.283		0.264	0.032
			Reference Material CLV-2			
LAB- 1	0.03	0.03	0.03	0.03	0.030	0.000
LAB- 2	0.04	0.03	0.03	0.03	0.033	0.005
LAB- 3	0.030	0.0223	0.0264	0.0242	0.026	0.003
LAB- 4	0.02	0.07	0.02	0.02	0.033	0.025
LAB- 6	0.07	0.05	0.06	0.05	0.058	0.010
LAB- 8	0.0379	0.0273	0.026		0.030	0.007

Table 9 – Laboratory results, means, and standard deviations for uranium in CLV-1 and CLV-2 (µg/g)

Laboratory	Independent results					Mean	S.D.
				Reference Material CLV-1			
LAB- 1	84.9	86.9	86.4	86.8		86.25	0.93
LAB- 2	88.3	87.6	87.0	88.2		87.78	0.60
LAB- 3	89.6	93.0				91.30	2.40
LAB- 4	79.2	74.2	75.9	75.9		76.30	2.09
LAB- 4	95.5	94.1	93.8	92.9		94.08	1.08
LAB- 5	87.2	64.1	83.0	87.7		80.50	11.13
LAB- 6	87.	79.	92.	90.	90.	87.60	5.13
LAB- 8	85.6	85.6	88.9			86.70	1.91
				Reference Material CLV-2			
LAB- 1	3.4	3.5	3.3	3.4		3.40	0.08
LAB- 2	3.4	3.6	3.8	3.7		3.63	0.17
LAB- 3	3.3	3.5				3.40	0.14
LAB- 4	2.6	2.6	2.7	2.6		2.63	0.05
LAB- 4	3.86	3.64	3.64	3.24		3.60	0.26
LAB- 5	3.5	3.3	4.4	4.0		3.80	0.50
LAB- 6	5.9	4.5	4.1	4.3	4.8	4.72	0.71
LAB- 8	3.26	4.06	3.46			3.59	0.42

Table 10 – Laboratory results, means, and standard deviations for cesium-137 in CLV-1 and CLV-2 (Bq/g)

Laboratory	Independent results				Mean	S.D.
			Reference Material CLV-1			
LAB- 4	0.111	0.113	0.112	0.107	0.111	0.003
LAB- 7	0.0945	0.0950	0.0979		0.096	0.002
LAB- 8	0.113				0.113	1
			Reference Material CLV-2			
LAB- 4	0.0391	0.0353	0.0367	0.0359	0.037	0.002
LAB- 7	0.0303	0.0334	0.0274		0.030	0.003
LAB- 8	0.0374				0.037	1

Table 11 – Radon-222 emanation efficiency, % E, of CLV-1 and CLV-2 (70 g/125 mL in steel can with open top, 72 cm²)

	Retained Rn-222[1], Bq g (±2σ)	% E[2](±2σ)
CLV-1	0.1827 ± 0.0042, 0.1785 ± 0.0042	73.7 ± 0.5
CLV-2	0.0274 ± 0.0028, 0.0264 ± 0.0028	30.3 ± 7.4

[1]Duplicate measurements 1 and 5 d after removal from bottle.
[2]100(1 − Rn-222 Ra-226), with Ra-226 gamma-ray determination.

Table 12 – Expected decay of lead-210[1] and cesium-137[2] activities in CLV-1 and CLV-2.
A/Ao is the predicted activity after t years relative to that at April 1985.

	A/Ao			
	CLV-1		CLV-2	
t (y)	Pb-210	Cs-137	PB-210	Cs-137
2	0.956	0.955	0.960	0.960
5	0.894	0.891	0.904	0.891
10	0.803	0.793	0.822	0.793

[1]$A/Ao = \exp(-\lambda t) + (1 - x)(A_{Ra}/Ao)[1 - \exp(-\lambda t)]$, where $\lambda = 0.0311\ y^{-1}$, and Ao and A_{Ra} are the measured Pb-210 and Ra-226 activities, respectively.
[2]Half-life of 29.94 y assumed.

APPENDIX A
REPORTS BY CONTRACTORS

APPENDIX A

Reports submitted by participants under contract completed March 1985*

Laboratory	Contract **		
	Title	Requisition No.	Serial No.
Dr. Colin Dunn Biogeochemist Regina, Sask.	Collection of vegetative material for use in a biological reference material project	23241-4-1681	OSQ84-00114
Chemex Labs Ltd. North Vancouver, B.C. with sub-contract to Saskatchewan Research Council Saskatoon, Sask.	Radiochemical analysis of vegetation reference materials CLV-1 and CLV-2	23241-4-1703	OSR84-00216
Monenco Analytical Laboratories Calgary, Alta.	Radiochemical determinations in vegetative reference materials II	23241-4-1704	OSR84-00217

* *Reports filed with the National Uranium Tailings Program, Canada Centre for Mineral and Energy Technology, 555 Booth Street, Ottawa, Canada, K1A 0G1.*

***Funded under the National Uranium Tailings Project.*

APPENDIX B
PARTICIPATING LABORATORIES

APPENDIX B

Participating Laboratories in Interlaboratory Program

Atomic Energy Control Board of Canada
Analytical Laboratory
D.F. Sullivan

Canada Centre for Mineral and Energy Technology
Mineral Sciences Laboratories
(a) *J.L. Dalton, R.H. McCorkell and M. Desgagné*
(b) *C.W. Smith*

Chemex Labs Ltd.
North Vancouver, B.C.
M. LeGeyt and E. Savoy

Department of Fisheries and Oceans
Freshwater Institute
Winnipeg
P. Wilkinson

Eldorado Resources Ltd.
Tunneys Pasture Analytical Laboratory
M.C. Miedma

Monenco Analytical Laboratories
Calgary
J. Dean and N. Chiu

Ontario Hydro
Pickering
G.J. Pilar

Saskatchewan Research Council
Analytical Services, Saskatoon
G. Smithson, D. Chorney, S. Magon and L. Turple

Slowpoke Reactor Facility
University of Toronto
R. Hancock

APPENDIX C

SUMMARY OF ANALYTICAL METHODOLOGY

APPENDIX C

Summary of radiochemical procedures for lead-210

	Lab 1	Lab 2	Lab 3	Lab 4a	Lab 4b	Lab 8
Decomposition	Acid digestion					Acid digestion + $K_2S_2O_7$ fusion
Separation	DDTC extraction of Bi-210				Direct gamma-ray	IX
Collection	BiOCl coprecipitation				Determination of Pb-210	Spontaneous deposition of Po-210
Counting	Beta-counting for Bi-210					Alpha counting
Yield	Bi by AA			Bi by XRF		Pb by AAS

Radiochemical procedures for polonium-210

All four laboratories used acid digestion and lab 8 also used a low-tempera- ture fusion. This was followed by spontaneous deposition of polonium onto silver or nickel discs. Alpha spectroscopy/ counting was used for measurement. Labs 3 and 8 used Po-208 and Po-209 respectively for yield measurement.

Summary of radiochemical procedures for radium-226

	Lab 1	Lab 2	Lab 3	Lab 4a	Lab 4b	Lab 5a	Lab 5b	Lab 8
Decomposition	$K_2S_2O_7$ fusion	Ashing + multiacid digestion	$HClO_4$ digestion	Ashing + multiacid digestion	Sample sealed in can		Multiacid digestion	Ashing, multiacid + $K_2S_2O_7$ fusion
Separation	$PbSO_4$ coprecipitation						$PbSO_4$ coprecipitation	Radon collection in charcoal trap
Collection	$BaSO_4$ coprecipitation						$BaSO_4$ coprecipitation	
Counting	Alpha spec/ gross alpha	Alpha spec			Gamma ray		Alpha spec	Radon emanation
Yield	Ba-133						Ba-133	

Summary of radiochemical procedures for thorium-230

	Lab 1	Lab 2	Lab 3	Lab 4	Lab 6	Lab 8
Decomposition	$K_2S_2O_7$ fusion	Ashing + multiacid digestion	$HClO_4$ digestion	Multiacid digestion	Multiacid digestion	Ashing, multiacid + $K_2S_2O_7$ fusion
Separations	$BaSO_4$ coprecipitation	$La(OH)_3$ coprecipitation	$PbSO_4$ coprecipitation + SX	IX	$BaSO_4$ coprecipitation	IX
Collection	$Th(OH)_n$ coprecipitation	LaF_3 coprecipitation	$Ce(OH)_3$ coprecipitation	$Ti(OH)_n$ coprecipitation	$Ti(OH)_n$ coprecipitation	SX with TTA
Counting	Alpha Spec					
Yield	Th-234	–	–	None	Th-234	Th-228

Summary of radiochemical procedures for uranium

Lab 1	Lab 2	Lab 3	Lab 4a	Lab 6	Lab 5	Lab 4b	Lab 8
	Delayed neutron counting after reaction irradiation		Multiacid digestion followed by optical fluorimetry		Multiacid digestion followed by laser fluorimetry	Gamma ray measurement of Th-234	Multiacid digestion IX & SX collection & separation alpha spec for U isotopes

Summary of radiochemical procedures for cesium-137 and potassium-40

Laboratories 4b, 7, and 8 used direct gamma-ray spectroscopy.